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MTL TR 89-63

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STRAIN AGING IN TUNGSTEN-HEAVY ALLOYS

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MATERIALS PRODUCIBILITY BRANCH

July 1989

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US ARMY
LABORATORY COMMAND
MATERIALS TECHNOLOGY LABORATORY

U.S. ARMY MATERIALS TECHNOLOGY LABORATORY
Watertown, Massachusetts 02172-0001

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER MTL TR 89-63	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) STRAIN AGING IN TUNGSTEN-HEAVY ALLOYS		5. TYPE OF REPORT & PERIOD COVERED Final Report
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Robert J. Dowding and Kenneth J. Tauer		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Army Materials Technology Laboratory Watertown, Massachusetts 02172-0001 SLCMT-MEM		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS D/A Project: P612105.H84
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Laboratory Command 2800 Powder Mill Road Adelphi, Maryland 20783-1145		12. REPORT DATE July 1989
		13. NUMBER OF PAGES 9
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Aging (materials)
Tungsten alloys
Heat treatment
Heavy metals

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

(SEE REVERSE SIDE)

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ABSTRACT

It has been shown that strengthening of tungsten-heavy alloys is possible through the use of cold working and heat treatment. The source of the increased strength has been attributed to strain aging due to the presence of carbon. Further, it was shown that this increase in strength did not have to be at the expense of elongation. For a 90% tungsten-heavy alloy, the strength increased from 164.7 ksi in the as-swaged condition to 178.4 ksi when heat treated at 700°C for 1 hour. Under the same conditions, the elongation increased from 10.8% to 12.2%. Similar results were observed for a 93% tungsten-heavy alloy. The strength increased from 173.9 ksi as-swaged, to 197.0 ksi when heat treated for 1 hour at 600°C. The elongation fully recovered at 800°C, held for 1 hour.

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INTRODUCTION

Tungsten-heavy alloys are two-phase mixtures of body-centered cubic (BCC) tungsten surrounded by a face-centered cubic (FCC) matrix.[1] The matrix is most often composed of nickel and iron in a ratio of 70:30 but, occasionally, the matrix may also contain cobalt or copper. Nickel, however, is always the primary component.[2-5] The tungsten-heavy alloy is fabricated through powder metallurgy (PM) techniques. Elemental powders are blended, pressed to shape, and sintered. Depending upon the tungsten content, the sintering temperatures are usually in the range of 1450°C to 1525°C. These temperatures are high enough that, as a result, the matrix is at the liquid phase and the process is known as liquid phase sintering.[6] At the liquid phase temperature, the matrix becomes saturated with tungsten, but this does not change the FCC character of the matrix.[5,7] The sintering is usually done in a dry hydrogen atmosphere furnace in order to reduce the oxides on the tungsten powder surfaces and create clean, active surfaces which will enhance the adherence between the tungsten and the matrix.[8] The hydrogen atmosphere also creates the presence of excess dissolved hydrogen in the alloy. It has been shown that the hydrogen degrades the toughness and ductility of the heavy alloy.[9] A post-sintering vacuum heat treatment is generally required to insure that there is no residual hydrogen present.

The as-sintered tensile strength of a 90% tungsten, 7% nickel, 3% iron alloy (called 90 W) is in the range of 116 ksi to 136 ksi (800 MPa to 940 MPa).[10] The strength can be increased by cold working which is commonly performed by swaging or rolling, depending upon the geometry.[11,12] Swaging to reductions in area of 20% can result in tensile strengths of 180 ksi or more. As the strength increases, the elongation, which may originally have been 25% or more, decreases to less than 5%.[7] The cold working of the 90 W alloy results in the multiplication of dislocations within the matrix and the tungsten grains. The number of dislocations can increase from 10^6 cm^{-2} in an annealed alloy, to 10^{12} cm^{-2} in a heavily cold-worked one.[13] The interaction of these dislocations with each other, grain boundaries, solute atoms, and microstructural defects, give the strength increases. This is generally referred to as strain hardening.[14]

Subjecting a cold-worked alloy to heat initiates several competing mechanisms. At low temperature (compared to the recrystallization temperature), the dislocations become mobile

1. GERMAN, R. M., BOURGUIGNON, L. L., and RABIN, B. H. *Microstructural Limitations of High Tungsten Content Heavy Alloys.* Journal of Metals, August 1985, p. 36.
2. CHURN, K. S., and GERMAN, R. M. *Fracture Behavior of W-Ni-Fe Heavy Alloys.* Met. Trans. A., February 1984, p. 331-338.
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and begin to annihilate themselves at dislocation sinks. These sinks may be grain boundaries, or they may be dislocations of opposite sign. The effect is an overall decrease in the strength of the alloy. At the same time, interstitial solute atoms that are able to diffuse at a fast enough rate, cause pinning of the dislocations and constrain their further movement. Additional heating will lead to the release of the stored energy of cold work, polygonization, and the formation of subgrain boundaries. Lastly, further heating will lead to recrystallization.[15]

Strain Aging

Interactions of solute atoms with dislocations during or after plastic working is known as strain aging.[16] In body-centered cubic metals, it is a phenomenon caused by the diffusion of interstitial solute atoms. The solute diffuses to the dislocation sites where the lattice is already strained. The effect is to reduce the lattice strain, due to the interstitials, and decrease the energy of the system.[16] The presence of these solute atoms at dislocations can cause a discontinous yield point, and the return of a yield point in previously strained metals, by locking the dislocations. Other observable changes due to strain aging are an increase in the yield point, an increase in the ultimate tensile strength, and a decrease in the elongation.[17] The interstitial solute atoms are usually carbon or nitrogen, but oxygen and hydrogen can also pin dislocations. Strain aging is a thermally activated process that is either static or dynamic. It can take place during a heat treatment or the thermal processing that may follow plastic working (static), or it can occur while working is taking place, usually at an elevated temperature (dynamic).[17] For static strain aging, the temperature must be high enough and the time must be long enough, but there are an infinite number of combinations of time and temperature that will result in strain aging. The only requirement is that the combination of time and temperature must be such that the interstitial atoms are able to diffuse to the dislocations. For dynamic strain aging, there is the additional factor of strain rate; the rate at which deformation is taking place. During dynamic strain aging, the solute atoms must diffuse fast enough to travel with the dislocations.

The strain aging of rimmed, low carbon steel is well known.[17,18] Strain aging has been observed at the heat-affected zone (HAZ) of welded sheet and plate that had previously been formed. It has also been found in steel that has been punched or sheared and then hot-dip galvanized. These observations have usually been the result of structural failures of one sort or another. Despite the failures, strain aging can be an inexpensive and effective method of strengthening metals when it is properly understood and exploited.

Strain aging has also been observed in "pure" tungsten as the return of the yield point in previously strained specimens.[19] It was determined that tungsten containing carbon would show strain-aging characteristics when there was a minimum, critical amount of carbon. Whereas eight parts per million (ppm) was not sufficent, 40 ppm resulted in a yield point return. In addition, a minimum prestrain of 7.5% was necessary. The temperature at which the yield point returned was between 650°C and 815°C. It can be concluded that there is a minimum set of conditions which must be fulfilled for strain aging to be seen. Northcutt, et al.,[11] observed what was considered strain aging in a 95 W-3.5 Ni-1.5 Fe alloy which had been

15. VERHOVEN, J. D. *Fundamentals of Physical Metallurgy.* John Wiley and Sons, Inc., NY, 1975, p. 325-361.
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cold rolled to a reduction in thickness of 30%. The Vickers microhardness (VHN) of the tungsten particles was seen to reach a maximum of 597 VHN at an annealing temperature of 600°C for times longer than 45 minutes. Yodogawa[7] examined the effects of cold rolling and annealing on a 90 W alloy. For reductions greater than 30%, the ultimate tensile strength (UTS) peaked at an annealing temperature of 500°C; for a 15% reduction, the peak was at 600°C, and a 5% reduction peaked at an annealing temperature of 800°C. The maximum UTS observed was for the 90% cold-rolled sample annealed at 500°C; it was approximately 300 ksi. The reductions in area of the tensile specimens dropped significantly from greater than 20% to less than 2%. The changes in strength with cold work and annealing were attributed to strain aging of the tungsten phase and precipitation hardening of the matrix phase that became saturated by tungsten during liquid phase sintering.

EXPERIMENTAL PROCEDURE

Tungsten-heavy alloys, 90 W and 93 W (90% W-7% Ni-3% Fe and 93% W-4.9% Ni-2.1% Fe, respectively), that had been swaged 18%, were obtained for strain-aging evaluation. Samples of each alloy received 1 hour isothermal anneals at 50°C increments, in vacuum. The range of temperatures was from 400°C to 1400°C. The evaluation of the optimum temperature for strain aging was performed by measuring the Vickers microhardness of single-tungsten grains. A minimum of three impressions were made for each heat treatment condition with a 100-gram test load. Using the optimum temperature, as determined by the microhardness tests and two temperatures near it, tensile specimens for both alloys were heat treated. These specimens, three for each condition, were tested at room temperature at a strain rate of 0.005/min. These specimens were used to determine the tensile response to strain aging of these alloys.

RESULTS AND DISCUSSION

Figure 1 is a plot of the Vickers hardness of individual tungsten grains versus the strain-aging temperature. It is clear that for either 90 W or 93 W, the hardness increases from an as-swaged value of approximately 438 VHN to a maximum of 531 VHN when aged at 800°C. Also, it is notable that the hardness, with minor exceptions for either alloy, is coincident at all temperatures. This could be expected since only the tungsten grains were tested and both alloys had the same processing history. Also in Figure 1, there is a plateau between the temperatures 600°C and 800°C where the hardness is somewhat independent of the temperature. Based upon the observation of this plateau, tensile samples of each alloy were heat treated. The results of the tensile testing will be discussed below. Stephens and Form[19] noted strain aging in "pure" tungsten in the temperature range 1200°F to 1500°F (650°C to 815°C). It was observed as the return of the yield point in samples that had been previously strained 7.5% (tensile). In that work, carbon was intentionally introduced to the tungsten in concentrations of 8 ppm and 40 ppm. It was discovered that, whereas 8 ppm was insufficient to cause the strain aging, 40 ppm caused the yield point to return upon proper heat treatment. Stephens and Form were also able to calculate the activation energy for the return of the yield point. Their value was 50.4 kcal/mole. It was very similar to published values of the activation energy for diffusion of carbon in tungsten, 53.5 kcal/mole.[20] They concluded that the strain aging of tungsten was a result of the presence and diffusion of interstitial carbon.

20. ASKILL, J. *Tracer Diffusion Data for Metals, Alloys and Simple Oxides.* IFI Plenum Data Corporation, 1970, p. 53.

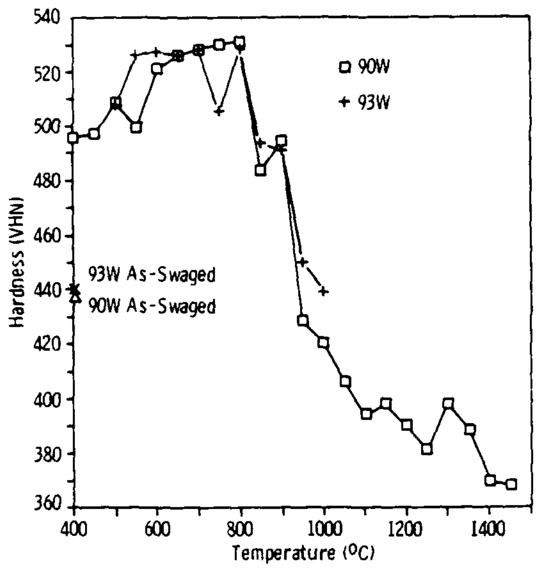

Figure 1. Hardness versus aging temperature (VHN versus degrees centigrade).

The microhardness measurements of this study show very similar results to that of Yodogawa[7] and Northcutt[11] in that, for 18% swaged 90 W, the hardness peaked in the range of 600°C to 800°C. The temperature that gives the peak is slightly higher than the one observed by Yodogawa, but that is in keeping with the lower amount of cold work imparted to the material.

Strain aging in tungsten-heavy alloys has been observed by Northcutt, et al.,[11] who showed that strain aging occurred in 95 W-3.5 Ni-1.5 Fe at temperatures from 500°C to 700°C. The material used by Northcutt had been cold rolled 30%, which is more cold work than used here or in the work previously cited. The tungsten grain hardnesses increased from an as-rolled value of 349 diamond pyramid hardness (DPH; same as VHN) to 597 DPH when aged at 600°C for 2 hours. The higher strain-aged hardness is likely due to greater strain hardening which is a direct result of the increased cold reduction. Northcutt, unsure of the cause, attributed the strain aging to carbon or hydrogen.

Yodogawa[7] performed a systematic study of a 90 W alloy in which the cold rolling was varied from 0% to 90%, and the annealing temperatures were from 200°C to 1420°C. It was seen that the tensile strength was maximized at 500°C and was independent of reduction except at reductions of 15% and lower. This is presumably due to a saturation of the dislocation density at the higher reductions. This would cause the diffusion distances necessary for pinning of the dislocations to be relatively constant at those higher reductions and, hence, become independent of the amount of work. It could be seen that at reductions of 15% and under, it required higher temperatures to bring about the tensile strength peak. Yodogawa also measured the microhardness of tungsten particles that had been cold rolled 30% and 50%, and found it reached a maximum at approximately 700°C. The increases in tensile strength and hardness observed by Yodogawa were attributed to strain aging of the tungsten and precipitation hardening of the matrix.

Samples were heat treated for tensile testing based upon the observations made in Figure 1. The 90 W samples were heat treated at 700°C, 750°C, and 800°C for 1 hour. The 93 W alloy was heat treated at 600°C, 700°C, and 800°C, also for 1 hour. The results of the tensile tests are shown in Table 1.

Table 1 TENSILE TESTING RESULTS

Alloy	Temperature	UTS (ksi)	0.2% YS (ksi)	Elongation (%)
90 W	As-Swaged	164.7	154.7	10.8
	700°C	178.4	158.1	12.2
	750°C	171.4	154.3	11.5
	800°C	170.2	151.3	12.4
93 W	As-Swaged	173.9	169.2	7.6
	600°C	197.0	189.3	5.1
	700°C	191.8	179.9	6.0
	800°C	181.5	169.3	7.5

The results of these tests are significant in that the tensile strength increased with heat treatment, and the elongation was retained or improved. The maximum tensile strength for the 90 W alloy occurred when aged at 700°C for 1 hour. The elongation was maximized over the as-swaged condition when heat treated at 800°C for 1 hour, increasing from 10.8% to 12.4%. Similarly, for the 93 W alloy, the tensile strength peaked at 600°C and the elongation recovered to the as-swaged value at 800°C. Above 800°C, as shown in Figure 1, the tungsten grain hardness drops off precipitously. This effect is likely due to recovery of the tungsten grains, which is the initial stage of recrystallization. It has been previously shown that recrystallization of tungsten in a heavy alloy can occur at temperatures as low as 900°C depending upon the amount of prior work.[7,21] Because of this, heat treatments above approximately 800°C would be useless in terms of strain aging. At the strain-aging temperatures, there are at least three events occurring. The first is the strain aging of the tungsten which, in this instance, is most likely due to the presence of carbon. The second is the recrystallization of the matrix as the temperature increases.[7] This is responsible for the increase in the elongation. Third, there is the recovery of the tungsten grains at temperatures above 800°C. Advantage may be taken of strain aging by heat treating for the desired combination of increased tensile strength and retained elongation. Further, it is possible to increase the elongation with the tensile strength.

CONCLUSIONS

1. The microhardness of the tungsten grains in either 90% or 93% tungsten-heavy alloys that were swaged 18%, can be increased through strain aging. The microhardness was shown to increase from an as-swaged value of approximately 438 VHN to 531 VHN when heat treated at 800°C for 1 hour.

2. The strain aging that occurred was attributed to the presence of carbon. It was shown previously, by others, that carbon would cause strain hardening in the temperature range investigated here.

21. FRANTSEVICH, I. N., TEDOROVICH, O. K., and BAZHENOVA, L. G. *Recrystallization of Tungsten in Tungsten-Nickel-Iron Alloys.* Soviet Journal of Powder Metallurgy-Metal Ceramics, v. 6, 1967, p. 393.

3. The tensile strength of the 90 W alloy was shown to increase from an as-swaged value of 164.7 ksi to 178.4 ksi when heat treated at 700°C for 1 hour.

4. The tensile elongation for the 90 W alloy was shown to increase from 10.8% to 12.2% under the same conditions.

5. The tensile strength of the 93 W alloy was shown to increase from an as-swaged value of 173.9 ksi to 197.0 ksi when heat treated at 600°C.

6. The tensile elongation of the 93 W alloy was shown to recover to the as-swaged value when heat treated at 800°C for 1 hour.

7. The improvement in the elongation was attributed to recystallization of the matrix.

ACKNOWLEDGMENTS

The authors wish to thank Dr. James Mullendore of GTE Chemical and Metallurgical Division for the material used in this study. The authors also wish to acknowledge the contributions of the following people: Mr. Thomas Moynihan and Mr. Richard Colena for assistance in heat treating, Mr. Andrew Zani for preparing the metallographic samples, and Mr. Gary Pelletier for doing the mechanical testing.

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STRAIN AGING IN TUNGSTEN-HEAVY ALLOYS -
Robert J. Dowding and Kenneth J. Tauer

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Technical Report MTL TR 89-63, July 1989, 9 pp-
table-illus. D/A Project: P612105.H84

Key Words

Aging (materials)
Tungsten alloys
Heat treatment

It has been shown that strengthening of tungsten-heavy alloys is possible through the use of cold working and heat treatment. The source of the increased strength has been attributed to strain aging due to the presence of carbon. Further, it was shown that this increase in strength did not have to be at the expense of elongation. For a 90% tungsten-heavy alloy, the strength increased from 164.7 ksi in the as-swaged condition to 178.4 ksi when heat treated at 700°C for 1 hour. Under the same conditions, the elongation increased from 10.8% to 12.2%. Similar results were observed for a 93% tungsten-heavy alloy. The strength increased from 173.9 ksi as-swaged, to 197.0 ksi when heat treated for 1 hour at 600°C. The elongation fully recovered at 800°C, held for 1 hour.

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